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IS 6947-3 (1975): Methods of estimation of composite pigments in oil pastes, ready mixed paints and enamels, Part 3: Estimation of lead chromes, iron blue and zinc compounds [CHD 20: Paints, Varnishes and Related Products]



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IS : 6947 (Part III)-1975
(Reaffirmed 2009)

Indian Standard

METHODS OF ESTIMATION OF COMPOSITE PIGMENTS IN OIL PASTES, READY MIXED PAINTS AND ENAMELS

PART III ESTIMATION OF LEAD CHROMES, IRON BLUES AND ZINC COMPOUNDS

Second Reprint SEPTEMBER 1999
(Incorporating Amendment No. 1)

UDC 667.633 : 543 [667.622]

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAKAR MARG
NEW DELHI 110002

Indian Standard

METHODS OF ESTIMATION OF COMPOSITE PIGMENTS IN OIL PASTES, READY MIXED PAINTS AND ENAMELS

PART III ESTIMATION OF LEAD CHROMES, IRON BLUES AND ZINC COMPOUNDS

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Indian Standard
**METHODS OF ESTIMATION OF
COMPOSITE PIGMENTS IN OIL PASTES, READY
MIXED PAINTS AND ENAMELS**

**PART III ESTIMATION OF LEAD CHROMES, IRON
BLUES AND ZINC COMPOUNDS**

0. FOREWORD

0.1 This Indian Standard (Part III) was adopted by the Indian Standards Institution on 25 February 1975, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

0.2 While finalizing IS : 101-1964* for publication, the concerned Sectional Committee was confronted with the problem whether to include standard methods of tests for single pigments or not. It was decided that under clause 27.1.1.1 of IS : 101-1964* it may clearly indicated that analysis of single pigments shall be done as specified in individual specifications and analysis of composite pigments in accordance with a separate standard. Consequently, formulation of this standard was taken up. The Committee, however, agreed to issue the standard in parts. Consequently IS : 6947 (Part I)-1973† and IS : 6947 (Part II)-1975‡ were formulated. This standard (Part III) in the series prescribes methods of estimation of lead chromes, iron blues and zinc compounds.

0.3 This standard is a necessary adjunct to material specifications for oil pastes, ready mixed paints and enamels.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960§.

*Methods of test for ready mixed paints and enamels (*second revision*).

†Methods of estimation of composite pigments in oil pastes, ready mixed paints and enamels: Part I Estimation of lead, zinc oxide, titanium dioxide, calcium compounds and zinc sulphide.

‡Methods of estimation of composite pigments in oil pastes, ready mixed paints and enamels: Part II Estimation of zinc chromes, ferric oxide and aluminium.

§Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard (Part III) prescribes methods of estimation of lead chromes, iron blues and zinc compounds from composite pigments in oil pastes, ready mixed paints and enamels.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in 2 of IS : 101-1964* and IS : 1303-1963† shall apply.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (*see* 1070-1960‡) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. EXTRACTION OF PIGMENTS

4.1 Pigments from oil pastes and ready mixed paints shall be first extracted in accordance with the procedure given in 27 of IS : 101-1964*.

5. ESTIMATION OF LEAD CHROMES

5.0 General — Lead chromes estimation consists of determination of lead, chromium oxide and sulphur trioxide. For estimation of lead, two methods (one volumetric and the other gravimetric) have been prescribed. Volumetric method may be used for routine analysis and gravimetric method in case of any dispute.

5.1 Determination of Total Lead (Volumetric Method)

5.1.0 Outline of the Method — The pigment is treated with sodium hydroxide solution and filtered. The total lead is precipitated as the chromate with potassium chromate or dichromate solution in a medium buffered with a mixture of acetic acid and sodium or ammonium acetate. The precipitate is washed with water, dissolved in dilute hydrochloric acid and titrated iodometrically after adding potassium iodide with a standard solution of sodium thiosulphate.

*Methods of test for ready mixed paints and enamels (*second revision*).

†Glossary of terms relating to paints (*revised*).

‡Specification for water, distilled quality (*revised*).

5.1.1 Reagents

5.1.1.1 *Sodium hydroxide solution* — 10 percent (approximately).

5.1.1.2 *Sodium hydroxide solution* — 2 percent (approximately).

5.1.1.3 *Acetic acid solution* — 1 : 2 (v/v).

5.1.1.4 *Potassium chromate solution* — 4 percent (approximately).

5.1.1.5 *Sodium acetate solution* — 1 percent (approximately).

5.1.1.6 *Hydrochloric acid solution* — 1:1 (v/v).

5.1.1.7 *Potassium iodide, pure*

5.1.1.8 *Standard sodium thiosulphate solution* — 0.1 N.

5.1.1.9 *Starch solution* — 0.5 percent (approximately).

5.1.2. Procedure — Weigh accurately 0.3 to 1 g of the pigment as prescribed in Table 1 in a 250-ml beaker, add about 100 ml of sodium hydroxide solution (10 percent) and break up the agglomerated particles by stirring well with a glass rod. Boil slowly for 4 to 5 minutes over a wire gauze and place the beaker covered with a clock glass on a water-bath for 10 to 15 minutes. Filter over (liter paper (Whatman No. 30) in a 400-ml beaker and wash the residue, if any, twice by decantation with 20 ml portions of hot sodium hydroxide solution (2 percent;). Transfer the residue to the filter and wash well with hot water till the residue is free from chromate. Cool the filtrate, neutralize with acetic acid solution and add 10 ml of acetic acid solution in excess. (At this stage yellow precipitate of lead chromate will appear.) Make the volume of the solution to 150 to 200 ml with water and heat to boiling. Add drop by drop 10 ml of potassium chromate solution and stir constantly with a glass rod during addition of the reagent solution.

5.1.2.1 Boil for 5 to 10 minutes and then place the beaker on the water-bath for 15 to 30 minutes. Filter the lead chromate precipitate through sintered glass crucible (No. 4). Transfer the precipitate to the crucible and wash with sodium acetate solution till free from soluble chromates. After cooling, dissolve lead chromate in about 20 ml of cold hydrochloric acid solution (this 20 ml of cold hydrochloric acid solution should be added in 3 instalments 10 ml, 5 ml and 5 ml) under slow suction. Then wash the crucible 3 to 4 times with cold water. Transfer the solution to the same beaker and adjust volume with water to about 150 to 200 ml. Add 2 g of potassium iodide and titrate the liberated iodine with standard sodium thiosulphate solution using starch as an indicator (2 to 3 ml of starch solution are to be added towards the end of the titration).

NOTE — Lead chromate precipitate may also be filtered over a small-filter paper (Whatman No. 30) and the precipitate along with the filter paper transferred to the beaker and treated with 20 ml of hydrochloric acid solution for titration.

TABLE 1 QUANTITY OF PIGMENT TO BE TAKEN FOR VOLUMETRIC DETERMINATION OF LEAD

(Clause 5.1.2)

SL No.	DESCRIPTION	SPECIFICATION	QUANTITY OF PIGMENT (g)
(1)	(2)	(3)	(4)
i)	Lead chrome pigments for paints	IS : 50-1967*	0.25 to 0.35
ii)	Brunswick green pigment for paints:	IS : 53-19501†	
	a). Genuine		0.5
	b). Reduced		1.0
iii)	Oil paste for paints (yellow)	IS : 94-1950‡	0.25 to 0.35
iv)	Oil paste for paints (green)	IS : 91-1950§	1.0
v)	Ready mixed paint, brushing, finishing, semi-gloss, for general purposes (yellow & brown)	IS : 118-1962 IS : 121-1962¶	0.5 1.0
vi)	Ready mixed paint, brushing, finishing, semi-gloss, for general purposes (green)	IS : 119-1962@	1.0

NOTE — For the material conforming to specifications not mentioned in this table suitable amount of the pigment shall be taken for the test.

*Specification for lead and scarlet chromes (*second revision*).

†Specification for brunswick green for paints.

‡Specification for oil paste for paints to Indian Standard colours No. 355, 356, 368 and 557.

§Specification for oil paste for paints to Indian Standard colours No. 218, 220 to 227 and 267.

||Specification for ready mixed paint, brushing, finishing, semi-gloss, for general purposes, to Indian Standard colours No. 355, 356, 368 and 557 (*revised*).

¶Specification for ready mixed paint, brushing, finishing, semi-gloss, for general purposes, to Indian Standard colour No. 414 Golden brown (*revised*).

@Specification for ready mixed paint, brushing, finishing, semi-gloss, for general purposes, to Indian Standard colours No. 218, 220 to 227, 267 & 284 (*revised*).

5.1.3 Calculation

$$\begin{aligned} \text{Total lead expressed as Pb(),} \\ \text{percent by mass} \end{aligned} = \frac{V \times N \times 0.0743 \times 100}{M}$$

where

V = volume in ml of standard sodium thiosulphate solution required for titration,

N = normality of standard sodium thiosulphate solution used, and

M = mass in g of pigment taken for analysis.

5.2 Determination of Total Lead (Gravimetric Method)

5.2.0 Outline of the Method — The pigment is treated with hydrochloric acid and the total lead precipitated as sulphide with hydrogen sulphide in acid solution. The precipitate is dissolved in dilute nitric acid, the solution fumed with sulphuric acid and the lead sulphate precipitate, after filtration and drying, is weighed as PbSO_4 and total lead is calculated.

5.2.1 Reagents

5.2.1.1 Hydrochloric acid solution — 1:1 (v/v).

5.2.1.2 Hydrogen sulphide— gas.

5.2.1.3 Sulphuric acid — Relative density 1.84.

5.2.1.4 Ethyl alcohol — 90 percent by volume.

5.2.1.5 Nitric acid — Relative density 1.40.

5.2.1.6 Ammonia solution — 1:1 (v/v).

5.2.1.7 Dilute sulphuric acid — 3 percent by volume.

5.2.1.8 Hydrogen sulphide solution — Saturated.

5.2.2 Procedure — Weigh accurately 0.5 to 1 g of the pigment in a dry 400-ml beaker and incinerate it on a low flame over a wire gauze to destroy organic matter particularly in the case of prussian blue. Add about 60 ml of hydrochloric acid solution and 2 ml of alcohol, cover with a clock glass and boil slowly over a wire gauze for about 5 to 10 minutes (at this stage the colour of the solution is clear bluish green). Dilute to about 200 ml with hot water, filter and wash the residue, if any, and the filter paper (Whatman No. 30) with hot water until a few drops of the filtrate give no indication for lead with hydrogen sulphide solution. Nearly neutralize the filtrate with ammonia solution in presence of a piece of litmus paper and add 5 ml of hydrochloric acid solution. Make the volume of the solution to about 300 ml with water and pass hydrogen sulphide gas for about 15 minutes. Allow the precipitate to settle for 5 minutes and then filter (Whatman No. 30). Wash the precipitate by decantation once with hydrogen sulphide solution, transfer it to the filter paper and wash it again on the filter paper 4 to 5 times with hydrogen sulphide solution. Dissolve the precipitate in hot nitric acid, collect the solution in a 400-ml beaker and wash the filter paper 4 to 5 times with a jet of hot water, collecting the washings in the same beaker. Cool and add 10 ml of sulphuric acid. Evaporate the mixture on a hot plate or sand-bath until dense white fumes of sulphuric acid are evolved. Cool, dilute carefully with 100 ml of distilled water and add 100 ml of ethyl alcohol. Mix thoroughly and allow it to stand for at least four hours. Filter off the precipitate of lead sulphate through a weighed porcelain Gooch or sintered glass (No. 4) crucible. Transfer the precipitate to the filter by a jet of dilute sulphuric

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acid and finally wash it with ethyl alcohol until free from acid. Dry the crucible at 130°C in an oven for about half-an-hour. Cool in a desiccator and weigh. Repeat drying until constant mass is attained.

5.2.3 Calculation

$$\begin{array}{l} \text{Total lead (expressed as PbO),} \\ \text{percent by mass} \end{array} = \frac{73.6 \times M_1}{M_2}$$

where

M_1 = mass in g of the precipitate, and

M_2 = mass in g of the pigment taken for analysis.

5.3 Determination of Chromium Oxide

5.3.0 Outline of the Method—The pigment is treated with sodium hydroxide solution and filtered. The filtrate is acidified with hydrochloric acid and chromium oxide is determined by titrating the iodine liberated from potassium iodide with standard sodium thiosulphate solution.

5.3.1 Reagents

5.3.1.1 Sodium hydroxide solution—10 percent (approximately).

5.3.1.2 Sodium hydroxide solution—2 percent (approximately).

5.3.1.3 Hydrochloric acid solution—1:1 (v/v).

5.3.1.4 Zinc sulphate solution—10 percent (approximately).

5.3.1.5 Potassium iodide solution—10 percent (freshly prepared).

5.3.1.6 Standard sodium thiosulphate solution—0.1 N.

5.3.1.7 Starch solution—0.5 percent (approximately).

5.3.2 Procedure—Weigh accurately 0.4 to 1 g of the pigment in a 250-ml beaker, add about 100 ml of sodium hydroxide solution (10 percent) and break up agglomerated particles by stirring well with a glass rod. Boil slowly for 4 to 5 minutes over a wire gauze and place the beaker, covered with a clock glass, on a water-bath for about 10 minutes. Filter over a filter paper (Whatman No. 30) in a 400-ml beaker and wash the residue, if any, twice by decantation with 20 ml portions of hot sodium hydroxide solution (2 percent). Transfer the residue to the filter, wash well with hot water till the residue is free from chromate and cool the filtrate. If prussian blue is present in the pigment, add 10 ml of zinc sulphate solution to the filtrate. Neutralize with hydrochloric acid solution and add 20 ml of hydrochloric acid solution in excess. Add 30 ml of potassium iodide solution and allow to stand for 3 minutes in the dark. Titrate the liberated iodine with standard sodium thiosulphate solution using 5 ml of starch solution as indicator.

5.3.3 Calculation

$$\begin{array}{l} \text{Chromium oxide (CrO}_3\text{),} \\ \text{percent by mass} \end{array} = \frac{3.334 \times V \times N}{M}$$

where

V = volume in ml of standard sodium thiosulphate solution used,

N = normality of standard sodium thiosulphate solution used, and

M = mass in g of the pigment taken for the analysis.

5.4 Determination of Sulphur Trioxide

5.4.0 Outline of the Method—The pigment is treated with hydrochloric acid, boiled and filtered. The acidity is reduced and a dilute solution of barium chloride added slowly to the hot filtrate. The precipitate of barium sulphate is filtered off, washed with water, carefully ignited and determined gravimetrically.

5.4.1 Reagents

5.4.1.1 Hydrochloric acid—Relative density 1.16.

5.4.1.2 Ammonia solution—1:1 (v/v).

5.4.1.3 Ammonium acetate solution—10 percent.

5.4.1.4 Barium chloride solution—5 percent.

5.4.1.5 Silver nitrate solution—2 percent.

5.4.2 Procedure—Weigh accurately 0.5 to 1 g of the pigment in a dry 400-ml beaker and incinerate it on a low flame over a wire gauze to destroy the organic matter particularly in the case of prussian blue. Add about 50 ml of hydrochloric acid, cover with a clock glass and boil slowly over a wire gauze for 5 to 10 minutes. Reduce the volume of the acid solution to about 25 ml by evaporating it on the water-bath. Dilute to about 200 ml with hot water, neutralize with ammonia solution in presence of a piece of litmus paper and add 1 ml of hydrochloric acid. Boil for a minute and place the beaker, covered with clock glass, on the water bath for about two hours. Filter (Whatman No. 40) and collect the filtrate in a clean beaker. Wash the residue twice by decantation with about 10 ml portions of hot solution of ammonium acetate, transfer it to the filter paper and wash 4 to 5 times with a jet of hot water, collecting all the washings in the same beaker. Heat the filtrate to boiling, add dropwise from a pipette 20 ml of barium chloride solution and stir the solution constantly during the addition. Keep the covered beaker hot for about two

hours on a water-bath. Test the clear supernatant liquid with a few drops of barium chloride solution for complete precipitation. Filter through a filter paper (Whatman No. 30) and transfer the precipitate to the filter paper with the aid of a jet of hot water. Wash the precipitate (7 to 8 times) with small portions of hot water until about 5 ml of the wash solution gives no opalescence with one or two drops of silver nitrate solution. Transfer the precipitate with the filter paper in an ignited, cooled and weighed porcelain crucible. Dry the paper by placing the crucible on a triangle above a small flame and gradually increase the heat until the paper chars. Raise the temperature to dull redness and finally, when the precipitate is white, ignite the crucible at red heat for about 10 minutes. Allow the crucible to cool somewhat in air, transfer to a desiccator and when cold, weigh the crucible. Repeat ignition until constant mass is attained.

5.4.3 Calculation

$$\begin{array}{l} \text{Sulphur trioxide (expressed as SO}_3 \text{),} \\ \text{percent by mass} \end{array} = \frac{M_1 \times 34.3}{M_2}$$

where

M_1 = mass in g of the precipitate, and

M_2 = mass in g of the pigment taken for analysis.

5.5 Report — From the results of analysis as determined from 5.1 to 5.4 the amount of lead chromate shall be calculated.

6. ESTIMATION OF ZINC COMPOUNDS

6.0 Outline of the Method — The pigment is treated with acetic acid-ammonium acetate solution and filtered. Zinc oxide is determined in the filtrate by the iodometric (ferric yanide) method.

6.1 Reagents

6.1.1 Acetic Acid-Ammonium Acetate Solution — This shall be prepared by dissolving 50 ml of glacial acetic acid and 20 g of ammonium acetate in a litre of water.

6.1.2 Potassium Iodide Solution — 10 percent (freshly prepared).

6.1.3 Standard Sodium Thiosulphate Solution — 0.1 N.

6.1.4 Starch Solution — 0.5 percent.

6.1.5 Ammonia Solution — 1:1 (v/v).

6.1.6 Potassium Ferricyanide Solution — M/10. It shall be prepared in cold water, filtered rapidly and kept in a dark bottle. The solution can be suitably used for seven days if kept in dark when not in use.

6.1.7 Ammonium Bifluoride (NH_4) HF_2 — solid.

6.2 Procedure — Treat 1 g of the pigment, accurately weighed, in a 250-ml beaker with 100 ml of acetic acid-ammonium acetate solution, stir well with a glass rod for 5 minutes and allow it to stand for 3 hours or overnight. Filter over filter paper (Whatman No. 30) in a 500-ml conical flask and wash with 10 ml portion of diluted acetic acid-ammonium acetate solution (5 ml of acetic acid-ammonium acetate solution diluted to 50 ml with water) five times. The filtrate shall be colourless. The yellow colour of the filtrate shall indicate the presence of zinc chrome in the pigment. Add ammonia solution dropwise until the red litmus paper just turns blue. Redissolve the precipitated ferric hydroxide, if any, by ammonium bifluoride (adding pinch by pinch), cool the solution, add 2 to 3 g of ammonium bifluoride, (at this stage blue litmus shall turn red), 30 ml of potassium iodide solution and sufficient water to make volume approximately 250 ml. Add 2 to 3 ml of starch solution and titrate the liberated iodine with standard sodium thiosulphate solution. Note the volume of thiosulphate solution. This indicates the amount of chromate present as zinc chrome. Next add 5 ml of potassium ferricyanide solution. Shake the flask and keep it in dark for about 2 minutes. Then titrate the liberated iodine against standard sodium thiosulphate solution. At the end point, the colour will be bright pale greenish yellow. If there is no such colour except whitish precipitate, add a further quantity (4 to 5 ml) of ferricyanide solution and titrate the liberated iodine again.

6.3 Calculation

$$\begin{array}{l} \text{Zinc oxide (ZnO), percent} \\ \text{by mass} \end{array} = \frac{12.45 \times V \times N}{M}$$

where

V = volume in ml of sodium thiosulphate solution used,

N = normality of standard sodium thiosulphate solution used,
and

M = mass in g of pigment taken for the analysis.

6.3.1 If chromate is present, and it is desired to estimate equivalent chromium trioxide, calculate as given below:

$$\begin{array}{l} \text{Chromium trioxide (CrO}_3\text{),} \\ \text{percent by mass} \end{array} = \frac{3.334 \times V_1 \times N}{M}$$

where

V_1 = volume in ml of sodium thiosulphate solution used in the titration before addition of ferricyanide solution, and
and M have the same legend as given in 6.3.

7. ESTIMATION OF IRON COMPOUNDS

7.0 Outline of the method — The pigment is incinerated to destroy cyanogen compounds, ferric iron is dissolved in hydrochloric acid and reduced to ferrous state with stannous chloride solution. Iron is estimated by oxidimetric titration of ferrous solution using standard solution of potassium dichromate.

7.1 Reagents

7.1.1 Standard Potassium Dichromate Solution — 0.1 N.

7.1.2 Stannous Chloride Solution — Dissolve 12 g of pure tin or 30 g of crystallized stannous chloride in 100 ml of hydrochloric acid (relative density 1.16) and dilute with water to 200 ml.

7.1.3 Hydrochloric Acid — Relative density 1.16.

7.1.4 Dilute Sulphuric Acid — 2.5 percent by volume.

7.1.5 Phosphoric Acid — 85 percent.

7.1.6 Mercuric Chloride Solution — Saturated.

7.1.7 Diphenylamine Indicator — 1 percent solution in concentrated sulphuric acid.

7.2 Procedure — Weigh accurately about 1 g of the pigment in a dry 250-ml beaker and incinerate it on a low flame over a wire gauze to destroy the cyanogen compounds or other organic matter. Add about 30 ml of hydrochloric acid and 30 ml of water, cover with a clock glass and place on the water-bath for 3 to 4 hours or overnight. Then gently boil the contents of the beaker over a wire gauze for a few minutes to get the pigment (excepting barytes and other siliceous matter) into complete solution. Filter over filter paper (Whatman No. 30) into a 500-ml conical flask, wash with hot water and evaporate the solution to about 50 ml. Heat to boiling and add the solution of stannous chloride dropwise, with agitation after each addition, until the liquid has a clean bluish green colour, quite free from any tinge of yellow. Add two to three drops in excess, cool the solution rapidly, add about 20 to 25 ml of saturated solution of mercuric chloride and allow it to stand for 3 to 5 minutes when a slight silky white precipitate shall be obtained. Then add about 150 ml of dilute sulphuric acid, 3 ml of phosphoric acid and 3 to 4 drops of diphenylamine indicator. Titrate slowly and with constant stirring against standard potassium dichromate solution to the first permanent violet-blue colouration.

7.3 Calculation

$$\begin{array}{l} \text{Ferric oxide (Fe}_2\text{O}_3 \text{), percent} \\ \text{by mass} \end{array} = \frac{8 \times V \times N}{M}$$

where

V = volume in ml of potassium dichromate solution used,

N = normality of potassium dichromate solution, and

M = mass in g of pigment taken for the analysis.

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Panel for Organic Pigments, CDC 50: 1 : 2

<i>Convener</i>	<i>Representing</i>
SHRI S. H. DESAI	Blundrll Eomite Paints Ltd, Bombay
<i>Members</i>	
SHRI M. G. B. CHANDRAN	Suhrid Geigy Ltd, Ahmedabad
DR O. P. MITTAL (<i>Alternate</i>)	
SHRI G. C. DESAI	Goodlass Nerolac Paints Ltd, Bombay
SHRI K. V. KRISHNAN	Colour-Chem Ltd, Bombay
SHRI R. M. TILLU (<i>Alternate</i>)	
SHRI K. L. RATHI	Sudarshan Chemical Industries Pvt Ltd, Poona
DR SHAIKH MAHAMOOD	United Carbon India Ltd, Bombay

Panel for Extenders, CDC 50: 1 : 4

<i>Convener</i>	
SHRI M. VAITHYANATHAN	Goodlass Nerolac Paints Ltd, Bombay

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones: 323 0131, 323 3375, 323 9402 Fax :+ 91 11 3234062, 3239399, 3239362

E - mail: bisind @ del 2.vsnl.net.in Internet: <http://www.del.vsnl.net.in/bis.org>

Central Laboratory:

Telephone

Plot No. 20/9, Site IV, Sahibabad Industrial Area, Sahibabad 201010

91 -77 00 32

Regional Offices:

Central: Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002 323 76 17

*Eastern : 1/14 CIT Scheme VII, V.I.P. Road, Kankurgachi, CALCUTTA 700054 337 86 62

Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022 60 38 43

Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113 235 23 15

†Western : Manakalaya, E9, MIDC, Behind Marol Telephone Exchange, Andheri (East), MUMBAI 400093 832 92 95

Branch Offices:

'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001 550 13 48

‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road, BANGALORE 560058 839 49 55

Commercial-cum-Office Complex, Opp. Dushera Maidan, Arera Colony, Bittan Market, BHOPAL 4620160 72 34 52

62/63, Ganga Nagar, Unit VI, BHUBANESHWAR 751001 40 36 27

Kalai Kathir Building, 670 Avinashi Road, COIMBATORE 641037 21 01 41

Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001 91-28 88 01

Savitri Complex, 116 G.T. Road, GHAZIABAD 201001 91-71 19 98

53/5 Ward No.29, R.G. Barua Road, 5th By-lane, GUWAHATI 781003 56 65 08

5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001 320 10 84

E-52, Chitaranjan Marg, C- Scheme, JAIPUR 302001 37 38 79

117/418 B, Sarvodaya Nagar, KANPUR 208005 21 68 76

Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road LUCKNOW 226005 21 89 23

NIT Building, Second Floor, Gokulpat Market, NAGPUR 440010 52 51 71

Patliputra Industrial Estate, PATNA 800013 26 28 08

Institution of Engineers (India) Building 1332 Shivaji Nagar, PUNE 411005 32 36 35

'Sahajanand House' 3rd Floor, Bhaktinagar Circle, 80 Feet Road, RAJKOT 360002 26 85 86

T.C. No. 14/1421, University P. O. Palayam, THIRUVANANTHAPURAM 695034 32 72 15

*Sales Office is at 5 Chowringhee Approach, P.O. Princep Street, CALCUTTA 700072 27 10 85

†Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007 309 65 28

‡Sales Office is at 'F' Block, Unity Building, Narashimaraja Square, BANGALORE 560002 222 39 71